Low-range flowmeters for use with vacuum and leak standards

K. E. McCulloh, C. R. Tilford, C. D. Ehrlich, and F. G. Long
National Bureau of Standards, Center for Basic Standards, Gaithersburg, Maryland 20899

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Vacuum pressure standards of the orifice-flow type require known gas flows of 10^{-6} mol/s (10^{-2} atm cm^3/s at 0 °C) and less. Known gas flows can also be used to calibrate "standard" leaks by comparing the pressures generated when flows from the leak and the flowmeter are alternately passed through a constant conductance. Two constant-pressure, piston displacement flowmeters developed at the National Bureau of Standards are described that can generate flows between 10^{-6} and 10^{-10} mol/s with an estimated uncertainty of 0.8% to 2%. Comparisons of the flowmeters with alternate calibration techniques, and repeated low-range leak and vacuum gauge calibrations, have been used to confirm the estimated uncertainty and random errors of the flowmeter.

I. INTRODUCTION

Two areas of vacuum standards require the generation of very low flows: orifice-flow pressure standards and leak standards. The orifice-flow technique generates a calculable pressure differential by passing a measured flow of gas through a calculated conductance. Reference 1 describes one such standard. The nominal 10 l/s conductance used is typical for this type of device, as is the working range of 10^{-1} Pa (10^{-3} Torr) and below. This requires flow measurements for different gases with a range starting at 5×10^{-7} mol/s (10^{-2} atm cm^3/s at 0 °C), and extending down three or more decades. Leak measurements cover an even wider range, and Refs. 2 and 3 describe how the flow from a "standard" leak can be measured by direct comparison with that from a flowmeter.

Gas flow rate is defined as the number of molecules or moles of a particular gas species passing through a system per unit time. A basic gas flow measurement must start with the equation of state; for the level of accuracy required for these applications the ideal gas law suffices:

$$PV = NRT \tag{1}$$

with the usual convention of P as the pressure, V as the volume, N the number of moles, R the ideal gas constant, and T the absolute temperature. As a practical matter, primary flowmeters are generally realized by varying only the pressure or volume, i.e., the molar flow rate is

$$dN/dt = (V/RT)(dP/dt) \tag{2}$$

or

$$dN/dt = (P/RT)(dV/dt)\,. \tag{3}$$

We have chosen to use the latter, "constant pressure" technique as the basis for our design. A constant pressure flowmeter was first used with an orifice-flow pressure standard by Hayward and Jepsen[4] and subsequently by several other experimenters. Their use has been reviewed by Peggs.[5] Basically, constant pressure flowmeters regulate the flow of gas from a small volume through a leak valve into the vacuum system. The pressure and temperature of the gas in the volume are measured and the gas volume is decreased at a rate sufficient to maintain a constant pressure. The volume change is generally accomplished by driving a piston of known cross section into the gas volume through sliding seals. The volume change and flowrate can then be derived from the rate of advance of the piston. A variant of this technique employs a welded bellows as the variable volume element.

We describe here two constant pressure flowmeters. The first employs a piston with sliding seals and has been in use at the National Bureau of Standards (NBS) for several years over the range from 10^{-6} to 10^{-11} mol/s (2×10^{-2}–2×10^{-7} atm cm^3/s at 0 °C). The second uses an all-metal gas system employing a hydraulically driven welded bellows as a variable volume. Its range extends at least two decades lower than that of the first although its performance at the lowest flows has not yet been fully analyzed.

II. DESCRIPTION AND OPERATION OF PISTON FLOWMETER

The piston and sliding seal ("piston") flowmeter is presented schematically in Fig. 1, and an enlargement of a piston assembly is shown in Fig. 2. The two pistons of different diameter (1 in. and 1 cm) allow for a wider range of flow rates than a single piston. The pistons are tungsten carbide or chromium plated steel wire gauges, supplied with a dimensional tolerance of 0.0005% along their lengths. They are sealed by Teflon rings with an L-shaped cross section, backed up by rubber O rings, as shown in Fig. 2. All other seals in the system are metal, including the vacuum valve closures. The O ring behind the Teflon acts solely as an elastic element to maintain the critical sealing force—the seal must have a minimal leakage but still allow free passage of the piston. A second ring of the same design, the top one in Fig. 2, serves to maintain the alignment of the piston along its axis of motion. A bypass channel vents the volume between rings to the ballast volume. This minimizes the effects of seal leakage by maintaining a guard pressure on the back side of the seal close to the operating pressure of the flowmeter. A clamp at the end of the piston and a coupling rod, which allows for radial misalignment, connect the piston to an external micrometer screw through a welded bellows. The bellows serves to seal the guard pressure. The micrometer screw is connected to the linkage by a bearing

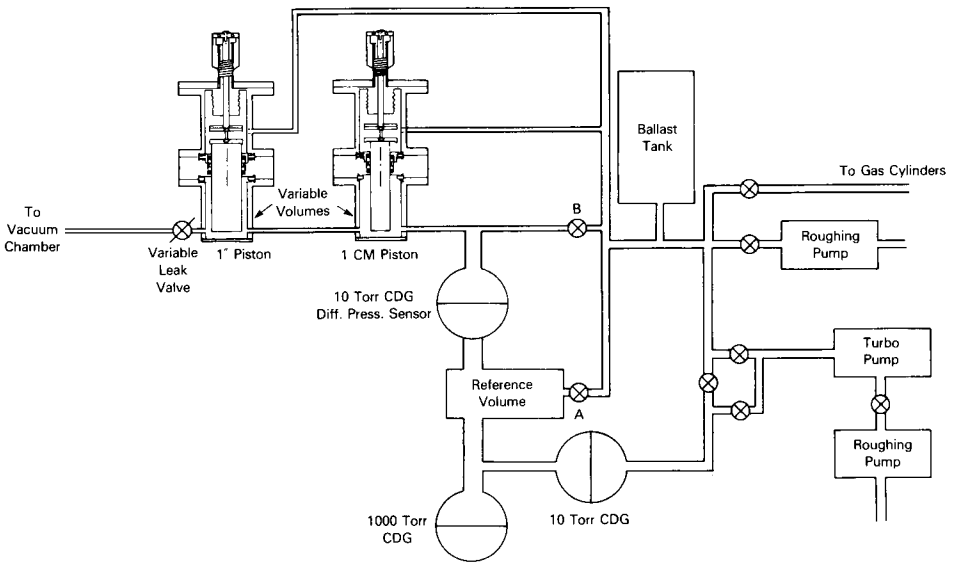

FIG. 1. Schematic of the constant pressure piston flowmeter. Two different size pistons are included to extend the range. The turbomolecular pump provides a low reference pressure for the 10-Torr differential capacitance diaphragm gauge.

which allows free rotation of the screw but maintains a tight axial coupling.

During operation the micrometer screw for the piston in use is advanced by a stepping motor at a rate that maintains a constant pressure in the flowmeter. A feedback circuit operating on the output of the 10-Torr differential capacitance diaphragm gauge (CDG) connected between the reference volume and the variable volumes controls the motor. The variable volumes, reference volume, and CDG are contained in a common thermally insulated area to minimize temperature induced pressure changes.

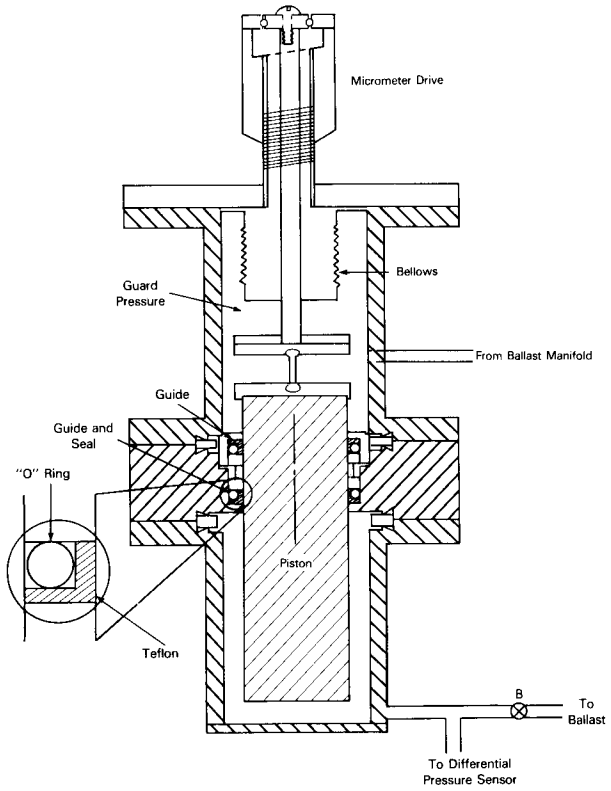

FIG. 2. Detail of the variable volume, including piston seal design and drive mechanism. All seals are metallic except for the sliding seal. To minimize leakage past the seal, a guard pressure close to the pressure in the variable volume is maintained on the back side of the seal by the line connected to the ballast manifold.

The pressure in the reference volume is measured with 10- and 1000-Torr CDG's calibrated against a primary standard.[6] As discussed below, instabilities in these gauges constitute the largest single source of error.

Gas flow to the vacuum chamber is regulated by an all-metal variable leak valve selected for stability and cleanliness. A 8000-cm^3 ballast tank allows a stable flow to be maintained for extended periods so that pressures in the vacuum chamber can be stabilized to within 0.1% before the flow is measured. After equilibration the pressure drops at a rate of 0.3%/h or less. Gas purity is monitored with a residual gas analyzer in the vacuum chamber. At the highest flowrates the indicated concentrations of residual gases are less than 0.01% of the test gas. It has not been determined whether these indicated impurities are in the original gas source or are generated in the gas analyzer ion source. In either case, they are below the level of concern. At lower flow rates residual gases become more important, presumably because of outgassing. In particular, at the lowest flows the indicated concentration of hydrogen, probably evolving from stainless-steel components, may exceed that of the test gas.

A trapped mechanical rough pump and a 50 l/s turbomolecular pump allow the system to be evacuated before the test gas enters the system. The flowmeter is not baked for fear of damaging the elastomer sliding seals. The turbopump also provides an absolute reference pressure for the 10-Torr CDG and permits the zero setting of the 1000-Torr CDG.

Before each use, the system is pumped with the turbomolecular pump to a pressure $< 10^{-4}$ Pa, measured with a hot filament ionization gauge. The piston that is not being used is driven fully into the flowmeter to minimize the variable volume. The turbopump is then valved from the system and gas of the desired species is admitted to the variable volumes,

ballast tank, and reference volume. The flowmeters have been operated with hydrogen, helium, neon, nitrogen, and argon. The system pressure is brought up to the value required to generate the desired flow in the vacuum system. Typical fill pressures vary from 10^5 Pa for a 10^{-6} mol/s flow to 10 Pa for 10^{-11} mol/s. The variable leak valve is preset so that about 1000 s is required for the piston to advance over its 2.5-cm range. This procedure is found to give the smoothest operation of the piston drive system. The variable leak valve is typically set at least 5 h before operation to allow any creep in the valve to settle out. After filling the flowmeter with gas, the gas flow to the vacuum chamber is maintained until the pressure in the vacuum chamber and the absorption of gas in the sliding seals have equilibrated. Equilibration times of 30 to 60 min are typical.

When equilibrium is achieved, valve A between the reference volume and the ballast tank is carefully shut so that there is negligible pressure differential across the CDG between the reference volume and the variable volumes. Valve B between the variable volumes and the ballast tank is then shut, and the pressure in the variable volumes starts to drop as gas flows only from this relatively small volume. The drop in pressure in the variable volumes with respect to the reference pressure is detected by the CDG, whose output voltage is sent to a proportional motor control unit, which causes the stepping motor to turn, advancing the piston. The servo system maintains the differential pressure at less than 0.1% of the reference volume pressure for the lowest flow rates, and considerably lower at higher flow rates. The micrometer screw is allowed to travel 20 revolutions, advancing the piston 2.54 cm, with the elapsed time being logged after each revolution. After each run the variable volumes are again connected to the ballast volume, the piston retracted, and the cycle is ready to begin again.

III. DESCRIPTION AND OPERATION OF THE BELLOWS FLOWMETER

The hydraulically driven bellows ("bellows") flowmeter is shown schematically in Fig. 3. The basic principle of operation and much of the design are the same as those of the piston flowmeter. However, in this case the variable volume is a welded bellows, providing a bakable all-metal gas system that is virtually free of leaks and adsorption effects. Base pressures after baking are about 10^{-6} Pa. This permits operation at lower fill pressures and correspondingly lower flow rates. However, the volume change of the bellows is in all likelihood not linear with displacement. This problem is overcome with a technique used at the Electro Technical Laboratory in Japan.[7] The bellows is coupled to a piston by a sealed hydraulic reservoir surrounding the bellows. Care is taken to eliminate gas from the hydraulic reservoir so that as the piston is advanced through a sliding seal into the reservoir the volume of the bellows must decrease by a corresponding amount. Two pistons are used of the same size and type as in the other flowmeter. The sliding seals are of the same design but now must only seal oil. The bellows has a nominal cross section of 13 cm^2 and stroke of 2 cm.

The variable volumes, reference volume, and oil reservoir are contained in a common insulated aluminum block to minimize temperature gradients. The "dead" volumes in both the variable volumes and oil reservoir have been minimized.

A spinning rotor gauge (SRG) thimble and ball are connected to the variable volumes in anticipation of possible use for flowmeter control at very low fill pressures.

As shown in Fig. 3, the bellows is extended by a variable weight connected to the bellows through a small-diameter sliding seal. The weight used is such that an oil pressure

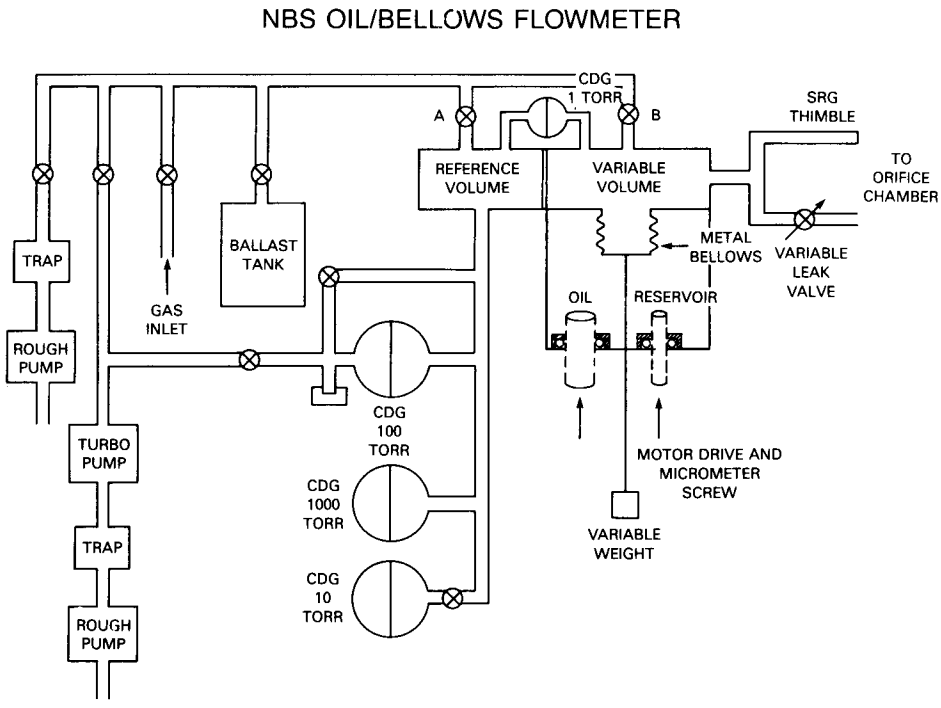

FIG. 3. Schematic of the constant pressure bellows flowmeter. The variable volume is a welded bellows coupled to two different diameter pistons by a sealed oil reservoir. The pistons are advanced by a micrometer screw and mechanism similar to that used in the piston flowmeter. All seals in the gas system are metallic.

slightly above atmospheric will be required to compress the bellows. This will minimize leaks, in particular, leakage of air into the oil, which would cause significant errors, and maintain positive contact between the piston and the drive mechanism. Procedures for the use of the bellows flowmeter are the same as those for the piston flowmeter.

IV. ESTIMATED FLOWMETER UNCERTAINTIES

The piston flowmeter has been in regular use for vacuum gauge and leak calibrations since 1984 and the bellows flowmeter since late 1985. During this time data have been collected on the uncertainties contributed by individual components of the flowmeters as well as on the overall performance. These data can be used to assess the probable errors of both flowmeters between 10^{-6} and 10^{-11} mol/s. The flowmeters have been operated at lower flows, particularly the bellows flowmeter, but further data are required to fully assess the uncertainties below 10^{-11} mol/s. The total uncertainty can be estimated by summing component errors.

From Eq. (1), the instantaneous flow rate can be derived as

$$dN/dt = d/dt[(PV)/(RT)]\,. \tag{4}$$

In practice an average flow rate $\overline{dN/dt}$ over a time Δt is measured:

$$\overline{dN/dt} = [(P_1V_1)/(RT_1) - (P_2V_2)/(RT_2)]/\Delta t\,, \tag{5}$$

where the subscript 1 indicates the respective measurement at the beginning of the time interval, and the subscript 2 indicates the respective measurement at the end of the time interval. In the case of the piston flowmeter this equation must be modified to allow for the possibility of gas leakage L past the sliding seal, and gas absorption or desorption D at the seal:

$$\overline{dN/dt} = [(P_1V_1)/(RT_1) - (P_2V_2)/(RT_2)]/\Delta t - L - D\,. \tag{6}$$

D and L are not known and cannot be corrected for, although their magnitude can be estimated and limits placed on the related errors.

Defining

$$\Delta T = T_2 - T_1\,, \tag{7}$$

$$\Delta V = V_1 - V_2\,, \tag{8}$$

and

$$\Delta P = P_2 - P_1\,, \tag{9}$$

Eq. (6) can be rewritten, to first order in ΔP and ΔT, as

$$\overline{dN/dt} = [(P_1\,\Delta V)/(RT_1\,\Delta t)]\{1 - (V_2/\Delta V) \times [(\Delta P/P_1) - (\Delta T/T_1)]\} - L - D\,. \tag{10}$$

In the constant pressure mode the measured flowrate is obtained from the first term of this equation, $P_1\,\Delta V/RT_1\,\Delta t$, and the remaining terms are regarded as sources of error. The estimated errors in the first term as well as those due to the other terms are tabulated at different flowrates for the piston flowmeter in Table I. The basis for these estimates is discussed below.

TABLE I. Uncertainties of the piston flowmeter in percent. The uncertainties of the bellows flowmeter are the same except at the lowest tabulated flowrate, where they are marginally smaller. Random errors are evaluated at three times the standard deviations or maximum observed deviations.

		Flowrate (mol/s) and piston size		
		10^{-6}–10^{-9} 1 in.	10^{-10} 1 cm	10^{-11} 1 cm
$\delta P_1/P_1$	systematic	0.50	0.50	0.52
	random	0.01	0.01	0.10
$\delta(\Delta V)/\Delta V$	area systematic	0.001	0.001	0.001
	length systematic	0.10	0.10	0.10
$\delta T_1/T_1$	systematic	0.003	0.003	0.003
	random	0.01	0.01	0.01
$\delta(\Delta t)/\Delta t$	random	0.01	0.10	0.10
$\frac{V_2}{\Delta V}\frac{\delta P_1}{P_1}$	systematic	0.20	1.30	1.30
$\frac{V_2}{\Delta V}\frac{\delta T_1}{T_1}$	systematic	0.006	0.04	0.04
Leakage	systematic	0.01	0.06	0.06
Systematic (Linear sum)		0.82	2.00	2.02
Random (rms sum)		0.02	0.10	0.14
Total		0.84	2.10	2.16

A. Initial fill pressure: $\delta P_1/P_1$

The systematic uncertainties are due almost entirely to observed long-term instabilities in the calibration of the capacitance diaphragm gauges used to measure the flowmeter fill pressure. Our experience with gauges used in the flowmeters has been consistent with our experiences with a larger group of gauges.[8] At the lowest flows there is an additional contribution due to a systematic offset in the servo system advancing the piston. The estimated random errors are due to random reading errors in the CDG's and the inability of the servo loop to maintain zero differential pressure between the reference and variable volumes. Modification of the servo system may reduce this error.

B. Volume change: $\delta(\Delta V)/V$

The uncertainties in piston cross-sectional areas are based on the specifications for the wire gauges. The uncertainties in the linear displacements are based on the maximum nonlinearities in the micrometer screws observed when checked against a standard length.

C. Initial temperature: $\delta T_1/T_1$

Systematic uncertainties are based on a maximum 10-mK error in the calibration of the quartz and platinum resistance thermometers used in the flowmeters. The random errors are based on observed temperature changes over 20–30 min periods, which are indicative of possible temperature differences between the gas and the thermometers.

D. Time interval: $\delta(\Delta t)/\Delta t$

The elapsed time is recorded for each turn as the micrometer screw advances. Time intervals are calculated from the differences of successive elapsed times. The first few of these 20 time differences may be excluded from the data analysis as they may be perturbed by small differential pressures generated during the closing of valve A and initial oscillations of the servo system driving the stepping motor. The times per turn exhibit random and systematic variations from turn to turn that depend on the fill pressure, and hence the flowrate. At higher fill pressures the times do not exhibit a systematic trend but have a random variation due to the 0.1-s resolution of the timing circuit. As the fill pressures are decreased the instabilities of the differential CDG and servo system become significant and the random variations in the time increase. The time interval Δt is an average of the individual times. However, with the piston flowmeter a systematic trend in the times becomes evident at 10^{-11} mol/s and becomes larger at lower flows. The times progressively decrease from the initial values towards an asymptotic value. The magnitude of the initial offset from the asymptotic value is roughly inversely proportional to the flowrate. This effect is substantially reduced for the bellows flowmeter. We believe this effect is caused by gas desorbed from the sliding seal as the piston moves through it. Therefore, we ignore those time intervals indicating a systematic trend and estimate an asymptotic value of the time interval from the remaining measurements. The probable error in estimating this asymptotic value is largely determined by the random variations of the individual times and the decreased number of "stable" time intervals that can be used in the estimate. Systematic errors in the timing circuitry are negligible.

E. Changes in the pressure during operation: $(V_2/\Delta V)(\delta P_1/P_1)$

We observe unexplained changes in the indicated pressure of the reference volume from the beginning to the end of the piston advancement. Their relative magnitude increases with decreasing fill pressures to a maximum of 0.1% at 10^{-11} mol/s. We do not believe these changes can be due to short-term CDG instabilities or temperature changes. We treat them as a possible additional error in the measured pressure of the variable volume. Their effect is multiplied by the ratio of the final or "dead" volume of the variable volumes to the volume change. This factor $V_2/\Delta V$ is 2 for the larger piston and 13 for the smaller. These pressure changes are almost always a pressure increase, therefore the associated uncertainty has been listed as systematic and evaluated on the basis of the maximum observed changes.

F. Temperature changes: $(V_2/\Delta V)(\delta T_1/T_1)$

Observed maximum 10-mK changes in the measured temperature are treated as possible changes in the gas temperature during the piston advancement. Again, the effect is larger for the smaller piston.

G. Leakage: L

Errors due to seal leakage in the piston flowmeter are estimated by deliberately establishing a large pressure differential across the seal and observing the rate of pressure change in the known variable volume. This observed leakage is scaled down to correspond to the small differential pressures across the sliding seal during operation.

H. Desorption: D

As noted earlier there is evidence at very low flowrates of gas desorption from the sliding seals in the piston flowmeter. Compensation is made for this effect by using asymptotic values of the time per revolution of the micrometer screw used to advance the piston. We believe the errors associated with this procedure are included in the increased random errors in the measurement of the time interval. Therefore, no separate uncertainty contribution for desorption is included in Table I.

From Table I it is apparent that improved accuracies for the flowmeters will largely depend on improvements in the pressure measurements. It is also important to minimize the dead or minimum volume V_2 of the variable volume. A large dead volume magnifies the effects of pressure or temperature instabilities.

In this same range the uncertainties of the bellows flowmeter will be substantially the same except that errors due to leakage or desorption have been eliminated.

The random errors listed in Table I are based on maximum observed errors. Therefore, the total error is computed from a linear sum of the systematic uncertainties plus a root sum of the squares of the random errors, with no multiplicative factor for the latter.

V. OBSERVED FLOWMETER PERFORMANCE

The validity of the uncertainty estimates in Table I can be experimentally checked to a certain extent. The estimated random error can be compared with observed random variations in the calibration of vacuum gauges or leak artifacts. Since the only errors common to both flowmeters are minor compared to the total, the errors of the two flowmeters are essentially independent of one another. Therefore, observed differences in the calibration of vacuum gauges or leak artifacts using the two flowmeters should be less than the linear sum of the errors of the two flowmeters. Finally, results obtained with these flowmeters can be compared with those obtained using other standards. In all cases the experimental data will include the random errors of the rest of the calibration system and the effects of instabilities inherent in the gauge or leak, and therefore, must be considered as an upper bound on the random errors of the flowmeters. This is particularly true of the low-range leak measurements. In these cases the pressures generated by the small helium flows are comparable to changes in the hydrogen background pressure in the vacuum chamber. This precludes the use of ion gauges to determine pressure equivalence of the flows from the leaks and the flowmeter. The quadrupole residual gas analyzers used at the low flows are significantly less stable than the ion gauges, shifts of several percent in sensitivity are not uncommon, and the random errors of the leak measurements are correspondingly higher.

A. Random errors

The measured random errors during gauge and leak calibrations differ with flowrate and type of device calibrated. The pooled standard deviation about the mean for six molecular drag gauges repeatedly calibrated over a two-month period using the piston flowmeter and flows between 2.2×10^{-9} and 1.3×10^{-7} mol/s did not exceed 0.3% for the piston flowmeter.[1] For flows of 10^{-9} mol/s the pooled standard deviation increased to 0.4%, but in this case the results are clearly limited by the imprecision of the molecular drag gauge. Repeated calibrations of helium diffusion leak at 5×10^{-11} mol/s, using both flowmeters, gave a standard deviation about the mean of 0.4% for six calibrations with the piston flowmeter and 0.5% for five calibrations with the bellows flowmeter. Repeated calibrations of a helium diffusion leak at 5×10^{-12} mol/s had standard deviations about the mean of 1.7% for 10 calibrations with the piston flowmeter and 1.0% for 17 calibrations with the bellows flowmeter.

These random errors clearly exceed those listed in Table I, but, as noted, they include instabilities of the leaks and gauges and random errors due to other parts of the calibration system.

B. Comparisons between flowmeters and with other standards

The means of molecular drag gauge calibrations performed with the piston and bellows flowmeter at 2×10^{-8} mol/s differed by 0.7%. The means of leak calibrations performed using the two flowmeters at 5×10^{-11} and 5×10^{-12} mol/s differed by less than the standard deviations of the individual flowmeter measurements so that any difference between flowmeters at this level is masked by the random errors of the individual flowmeters. The piston flowmeter has been used to calibrate molecular drag gauges that were calibrated using a "quantity" meter.[9] The results for four gauges differed by 0.1%, the results for a fifth gauge differed by 0.5%. This does not indicate a significant difference between calibrations performed using the piston flowmeter and those made using the quantity meter.

Calibrations of a sintered metal leak by the NBS and the Sandia National Laboratory Primary Standards Laboratory differed by no more than 1.5% between 10^{-8} and 10^{-6} mol/s.[2] There was evidence of a drift in the transfer leak, but the differences are, in any case, well within the combined uncertainties of the leak calibration standards of the two laboratories.

VI. SUMMARY

Two low-range, constant-pressure, piston displacement flowmeters have been developed at the NBS. The uncertainties have been evaluated in the flow range 10^{-6}–10^{-11} mol/s, and comparisons made with measurements performed on other systems or using other techniques in this same range. The estimated errors in the flow range 10^{-6}–10^{-9} mol/s of 0.8%, increasing to about 1.3% at 10^{-11} mol/s, appear consistent with the experimental results. Investigations are currently under way to assess the uncertainties at lower flowrates.

ACKNOWLEDGMENTS

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